

Quality Assurance Plan



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Frontier GeoSciences' policies and procedures are established in order to meet the requirements with the most current revision of the National Environmental Laboratory Accreditation Conference (NELAC) standards for Quality Systems.

A handwritten signature in black ink, reading 'Kristina Spadafora'.

Kristina Spadafora, Quality Assurance Officer

7/30/2008

A handwritten signature in black ink, reading 'Patrick Garcia Strickland'.

Patrick Garcia Strickland, Laboratory/Operations Manager, Lead Technical Director 7/30/2008

Frontier's Mission Statement:



Frontier GeoSciences is an advanced research and analytical laboratory specializing in mercury and trace metals characterization. Since 1992, our expertise in identifying the source, transportation, fate and effect of trace metals in atmospheric, geological, biological, and hydrological cycles has produced innovative and reliable analytical methods that are currently utilized around the world.

Private industry and government agencies alike rely upon Frontier GeoSciences to provide accurate and precise data so that they are able to make informed decisions about matters of serious human and economic consequence.

It is our mission to provide the highest quality results to solve complex environmental problems in the most scientifically sound and cost-effective manner.



CONTENTS

Quality
Assurance
Plan

Page 1

1	SCOPE	5
2	DEFINITIONS AND ABBREVIATIONS	6
3	QUALITY ASSURANCE STANDARDS AND POLICIES.....	8
3.1	Quality Assurance Policy Statement.....	8
3.2	Project Acceptance Policy	9
3.3	Ethics Policy	9
3.4	Financial Pressure Policy	10
3.5	Confidentiality Policy.....	10
3.6	Subcontracting Policy	10
4	LABORATORY ORGANIZATION AND RESPONSIBILITIES	12
4.1	Organization Chart.....	12
4.2	President	12
4.3	Operation Manager/ Laboratory Director, and Technical Director	13
4.4	Quality Assurance Officer	13
4.5	Office Administration.....	14
4.5.1	Accounting Manager and Human Resource Manager.....	14
4.5.2	Information Technology Services	14
4.6	Safety and Facility	15
4.7	Business Development	15
4.8	Technical Staff	15
4.8.1	Project Management Group	15
4.9	Mercury Supervisor.....	18
4.10	Hg Aquatic and Other Senior Analyst.....	19
4.11	Emissions Project Coordinator.....	19
4.12	MDN (Mercury Deposition Network) Site Liaison	20



4.13	Trace Metal Supervisor.....	21
4.14	Mercury Analyst.....	23
4.15	Trace Metals Analyst.....	23
4.16	Laboratory Technician.....	24
4.17	Shipping and Receiving Supervisor	25
4.18	Shipping and Receiving Laboratory Technician	25
5	LABORATORY CAPABILITIES AND SERVICES.....	27
5.1	Laboratory Certifications.....	27
5.2	Laboratory Experience	27
5.3	Field Sampling and Training.....	27
5.4	Analytical Capability	28
6	QUALITY ASSURANCE COMPLIANCE and OBJECTIVES	29
6.1	Proficiency Testing Program	30
6.2	Laboratory Inter-comparison Studies.....	30
6.3	Training Program.....	30
7	SAMPLE HANDLING	32
7.1	Sampling Procedures	32
7.2	Sample Acceptance.....	32
7.3	Sample Receipt	34
7.4	Chain of Custody	35
7.5	Sample Tracking.....	36
7.6	Tracking Specifics for the Mercury Group.....	36
7.7	Tracking Specifics for the Trace Metals Group	36
7.8	Mercury-Specific Sample Handling.....	36
7.9	Trace Metals Specific Sample Handling	37
7.10	Shipping of sample	37
7.11	Disposal.....	37



8	TRACEABILITY	38
8.1	Documentation.....	38
8.2	Reagents and Standards	38
9	TEST METHODS AND STANDARD OPERATING PROCEDURES	40
9.1	Test Methods and Standard Operating Procedures (SOPs).....	40
10	INTERNAL QUALITY CONTROL	45
10.1	Laboratory Quality Control Samples.....	45
10.2	Calibration of Analytical Instruments	45
10.3	Initial and Continuing Calibration Verification Standards (ICVs and CCVs)	46
10.4	Initial and Continuing Calibration Blanks (ICBs and CCBs)	46
10.5	Method Blanks.....	46
10.6	Certified Reference Materials (CRMs).....	46
10.7	Matrix Duplicate/Triplicate (MD/MT)	47
10.8	Matrix Spikes/Matrix Spike Duplicate (MS/MSD), Analytical Spike/Analytical Spike Duplicate (AS/ASD)	47
10.9	Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	47
10.10	Method Detection Limits.....	47
10.11	Method Validation.....	48
10.12	Control Charts	48
10.13	Data Reduction and Review	48
10.14	Report Format and Contents.....	49
11	PERFORMANCE AND SYSTEM AUDITS	51
11.1	Internal Laboratory Audits.....	51
11.2	External Laboratory Audits	51
11.3	Management Committee Review of Quality Assurance	51
11.4	Corrective Actions for NELAC policies.....	52
11.5	Incident Reports.....	52



11.6	Customer Complaints	52
12	DOCUMENT CONTROL AND MANAGEMENT	54
12.1	Document Management for Raw Data and Reports	54
12.2	Document Management for Training Files	54
12.3	Document Management for Controlled Documents	54
12.4	Document Management in the Event of Transfer of Ownership	54
13	FAUCILITIES INVENTORY AND CONTROL	55
13.1	Facilities and Equipment	55
13.2	Security	57
13.3	Computer Systems and Software	57
13.4	Bottle and Acid Vat Monitoring	58
13.5	Air Monitoring	58
13.6	Reagent Water Monitoring	59
14	REFERENCES	60



1 SCOPE

The purpose of the Frontier GeoSciences, Inc. Quality Assurance Plan (QAP) is to document the minimum quality assurance requirements for the laboratory. This Quality Assurance Plan provides ready reference for analysts, managers and clients on Frontiers policy pertaining to the accuracy and reliability of analytical tests performed in the laboratory.

The policies contained within this Frontier Quality Assurance Plan are to be applied to all company operations. The manual is updated when needed, to provide for the addition of new methods and procedures as they are developed.



2 DEFINITIONS AND ABBREVIATIONS

AS – Analytical Spike (sample spiked after extraction/digestion)

ASD – Analytical Spike Duplicate (sample spiked after extraction/digestion)

BLK – Method Blank

CAMR - Clean Air Mercury Rule

CCB - Continued Calibration Blank

CCV - Continued Calibration Verification

COC-Chain of Custody

CRM - Certified Reference Material

CV-AFS - Cold Vapor-Atomic Fluorescence Spectrometry

CV-GC-AFS - Cold Vapor – Gas Chromatography–Atomic Fluorescence Spectrometry

DOC - Demonstration of Capability

EDD - Electronic Data Deliverables

HAL – Mercury Analytical Laboratory

HG-AFS - Hydride Generation-Atomic Fluorescence Spectrometry

HG-CT-GC-AAS - Hydride Generation-Cryogenic Trapping-Gas Chromatography-Atomic Absorption Spectrophotometry

ICP-MS – Inductively Coupled- Plasma Mass Spectrometry

ICP-MS-DRC – Inductively Coupled-Plasma Mass Spectrometry-Dynamic Reaction Cell

ICB - Initial Calibration Blank

ICV - Initial Calibration Verification

IDOC – Initial Demonstration of Capability

LIMS - Laboratory Information Management System

MD - Matrix Duplicate MDL – Method Detection Limit

MDN - Mercury Deposition Network



MMO - Work Order Notes

MOF - Mercury Observer Form

MRL – Method Reporting Limit (=PQL)

MS - Matrix Spike (sample spiked prior to extraction/digestion)

MSD - Matrix Spike Duplicate (sample spiked prior to extraction/digestion)

MSDS - Material Safety Data sheets

MT – Matrix Triplicate

NAPD - National Atmospheric Deposition Program

NELAC - National Environmental Laboratory Accreditation Conference

NELAP - National Environmental Laboratory Accreditation Program

PB - Preparation Blanks

PE - Performance Evaluation

PQL - Practical Quantitation Limits

PT - Proficiency Test

QAP - Quality Assurance Plan

RPD - Relative Percent Difference

RSD – Relative Standard Deviation

SRM - Standard Reference Material SOP - Standard Operating Procedure

TAT - Turn Around Time

US EPA - United States Environmental Protection Agency



3 QUALITY ASSURANCE STANDARDS AND POLICIES

3.1 Quality Assurance Policy Statement

Frontier Geosciences has a vital commitment to Quality Assurance (QA), viewing it as both a program and a philosophy. We use quality control standards set forth in the NELAC regulations from the sample receipt, at the bench level, and continuously monitoring until sample results leave the laboratory. Our focus is on strong knowledge of the analytical procedures and adherence to strict enforcement of QA regulations. Prevention of analytical problems is accomplished by training. Frontier's laboratory staff is trained on how to troubleshoot and is encouraged to do so and to initiate corrective actions. Our management style is to solicit process improvement and problem solving from our technicians and analysts, then use management for implementation. This helps keep management informed and up to date, while at the same time promoting the professional growth of employees, and keeping our lab at the cutting edge of analytical technology. Frontier is dedicated to providing uncompromisingly high quality data that meets the needs of the environmental, geochemical, human health, and industrial communities.

Frontier recognizes that accurate and precise data depends on these basic principles:

- Sample integrity must be preserved. All documented sample handling procedures for preservation, custody, storage, labeling and record keeping are followed.
- Trace metal-free ("ultra-clean") sample handling must be employed. All samples to be analyzed for low level or ambient metals concentrations are handled according to established protocols. This includes the use of class-100 clean hoods, clean room gloves, and pretested and approved reagents, water, and equipment. Known high-level (contaminated) samples are kept segregated from ultra-clean samples during storage and sample preparation.
- Approved analytical methods must be followed. The analysts' fundamental understanding of analytical methods is paramount for effective quality control. Emphasis on scientific understanding and adherence to procedure is part of every analyst's training. Quality control results from each method are evaluated to identify and correct any method weakness, and to detect any need for further training.
- Analytical instrumentation must be in proper working order. Optimum instrument performance is ensured by analyzing daily calibration and quality control samples. Preventative maintenance is performed on a regular basis and is documented in instrument log books.
- Raw data must be properly reduced and accurately transcribed into the correct reporting format. Various levels of data review, from acquisition to the final report, are performed to minimize error.



- Precision and accuracy (bias) of analytical methods are documented and monitored and compared to historical data from reference materials.

3.2 Project Acceptance Policy

Project Managers are responsible for deciding whether or not to accept new work. When in doubt, prospective projects must be reviewed by the President or the Operations Manager. This generally pertains to large-scale or non-routine projects. Regarding all other new work, it is the sole responsibility of the Project Manager to evaluate whether or not it can be completed to meet the client's project goals, data quality objectives, and deadlines.

3.3 Ethics Policy

It is of paramount importance that all reported results and interpretations are objective and honest. Although individuals within the company may differ on the political implications of various results, Frontier must remain above this in its research and data reporting. All obtained results must be reported with complete honesty, with no regard for the expected, or preferred (by client or analyst) outcome.

In cases where the "best" or "most accurate" data have to be selected from an analytical set (or if data must be rejected), all criteria used in the evaluation must be clear and well documented in the LIMS MMO notes and communicated to the client with reasons and explanations in the report narrative. All documentation must be readily available for outside review. There may be times when the decision to reject data is not absolutely clear. Any time the decision is ambiguous, it must be made with management consent. Unambiguous decisions need not be overseen, or explained, but all data must be presented and noted on the original data set. Falsification of data or its deliberate suppression is considered grounds for immediate employment termination.

All personnel are required to adhere to Frontier's ethics policy. Any staff member witnessing an act that may violate the ethics policy is required to report the incident to management. The reporting staff member may maintain anonymity. Several avenues are available for staff to report suspect behavior: they may directly confront the offending staff member, they may report the instance to the staff member's direct supervisor, the Operations Manager, or any member of the Operations Management group. The important factors are for management to be aware of the situation, and for all personnel to feel comfortable reporting suspect behavior. When an employee brings up concerns of this nature, the Operations Manager or the Operations Management group must respond to the concern within 7 calendar days. The action shall be documented and kept on file in Human Resources (HR). HR is required to do a follow up after three months to ensure the concern from the employee has been resolved.

Violations of Frontier's ethics policy can negatively affect those involved, as well as the company as a whole. Individuals may face reputation damage, disciplinary action, job dismissal, legal action, or fines. Furthermore, the entire company may face a loss of business, legal action, fines, or even mandatory shutdown.



Ethics training is a requirement for each technical staff member. Our Ethics Training Program is administered by the QA Office and emphasizes the prevention of unacceptable activities. Training for all new staff members and a company wide annual data integrity refresher course is provided. Training is documented and kept on file in the QA Office.

3.4 Financial Pressure Policy

Frontier's primary purpose is to produce high quality, scientifically coherent data. In order to achieve this, the laboratory staff requires certain resources. One principal resource, commonly overlooked in routine laboratories, is time. To constantly improve the quality of their data, analysts at Frontier are encouraged to use their initiative and scientific intuition. Frontier's analysts can count on support for trouble-shooting and analyst-initiated repeat analyses because the production of sound scientific data is more important than the volume of samples analyzed. Therefore, analysts and technicians should distance themselves from any commercial, financial, and other undue pressures that may adversely affect the quality of their work. If staff members feel pressure in any of these areas, such that it is counter-productive to Frontier's analytical goals, they should bring it up with the Operation Manager or any member of the Operation Management group. When an employee brings up concerns of this nature, the Operations Manager or the Operations Management group must respond to the concern within 7 calendar days. The action shall be documented and kept on file in Human Resources (HR). HR is required to do a follow up after three months to ensure the concern from the employee has been resolved.

3.5 Confidentiality Policy

All of Frontier's controlled documents, inventions, ideas, processes, formulas, know-how, improvements, methods, designs, and techniques shall be treated as proprietary information and shall be protected by the Washington State Trade Secret Act, RCW 19.108 et seq., and other laws. Proprietary information shall be kept in the strictest confidence and shall not be used or appropriated for the benefit of any party without the prior written consent of Frontier.

Proprietary information shall not be summarized, copied, reproduced electronically or by any other method, and shall not be disclosed, distributed, disseminated or transmitted in any manner to any party without the prior written consent of Frontier. All proprietary information (including any originals, copies, summaries or other reproductions thereof) shall be and at all times remain the property of Frontier and shall be returned to Frontier upon demand.

It is also Frontier policy and responsibility to ensure the confidentiality of proprietary client information. Such information shall be released to a third party only with written permission of the client, except as required by law.

3.6 Subcontracting Policy

Project Managers will choose subcontracting laboratories based on reputability, client/project goals, and/or subcontracting agreements. If subcontracting agreements



are warranted, they will be developed specifically for the project in question, and subcontracting laboratories will be held responsible for guidelines/goals outlined in the agreement. The QA Office keeps all subcontracting laboratory certifications and company profiles in a file provided by the Project Manager. This file is available to all clients for review, except in cases where client confidentiality could be compromised.

At the Project Manager's discretion, laboratory services will be subcontracted for analyses outside our scope, and/or sample preparation requiring specialty instrumentation. Additionally, subcontracting services may be used on large-scale projects where instrumentation and/or staff size limitations require them. All subcontracting agreements will need prior client approval.



4 LABORATORY ORGANIZATION AND RESPONSIBILITIES

4.1 Organization Chart

A copy of Frontier GeoSciences is included in this Quality Assurance Plan as Appendix 1. The current version of the organization chart is located in:

FGS_Forms_Employee Documents\Organization Charts

Frontier GeoSciences is divided into the two analytical areas: Mercury and Trace Metals. Mercury has three sub groups: "Hg Aquatic and Other", "Emissions", and "MDN".

These departments have defined objectives and responsibilities as in the following descriptions. Analysts and technicians are cross trained and can perform job requirements in a many areas of the company which increases the analytical efficiency and sample throughput. Internal resources are distributed where they are most needed. This also increases the analysts' and laboratory technicians' knowledge of the overall process of the company.

Managers and supervisors from each department are represented in the Operations Management group, which has weekly "round table" meetings. Frontier GeoSciences strives to have a direct and open line of communication between groups, managers, and staff.

The managers provide supervision for department operations, implement the laboratory Quality Assurance Plan, ensure proper scheduling and execution of analyses, ensure that proper analysis techniques are being used (use of approved SOPs), supervise the review of all data, and report all discrepancies to the QA department.

The structure of Frontier GeoSciences provides a foundation for a high quality operation with the Quality Assurance Plan as its blueprint.

4.2 President

The President is ultimately responsible for financial management of the laboratory. The President is in direct and straightforward communication with the members of the Operation Management group, which include Operations Manager, Quality Assurance Officer, Safety & Facilities Manager, Accounting and Human Resources Manager, IT Manager Business Development Manager, Shipping and Receiving Supervisor, Project Management Supervisor, Research and Development Supervisor, Mercury Supervisor(s) and Trace Metals Supervisor. The President has the final say in implementation of corporate goals, objectives and policies.



4.3 Operation Manager/ Laboratory Director, and Technical Director

The Operations Manager/Laboratory Director reports directly to the President and is a member of the Operations Management group. The Operations Manager/Laboratory Director is referred to as the Operation Manager in this QAP. The Operations Manager works closely and supports all members of the Operations Management group, and provides up to date methods and information to the staff for all laboratory work. It is the Operations Manager responsibility to see that the non-laboratory departments (office administration, IT, and Business Development, etc.) of Frontier work with their laboratory counterparts to achieve high quality results.

For the purposes of NELAP compliance, a Technical Director must be named. The Operation Manager has the role as Technical Director. In the absence of the Operation Manager, the Deputy Technical Director role is filled by the Quality Assurance Officer, who will carry out the responsibilities required of that position.

4.4 Quality Assurance Officer

The Quality Assurance Officer reports directly to the President and is a member of the Operation Management group.

The QA officer communicates directly to the Operations Management group, analysts, technicians, and project managers to ensure that NELAC QA/QC requirements of this Quality Assurance Plan and of Frontier GeoSciences' Standard Operating Procedures (SOPs) are implemented and followed as written.

The QA Officer is responsible for the monitoring of daily laboratory QA/QC activities as follows:

- Reviews problematic data and a representative sample of standard level QA datasets to ensure a good quality system, and compliance with NELAC, contractual, or other regulatory requirements.
- Supervises the review of High QA client data packages and the writing of High QA data reports.
- Follows up on omissions and non-conformances and reports all non-compliance issues for correction. Maintains and follows up on internal incident reports and determine long and short term actions. Reports unacceptable findings to the Operation Manager and the President if needed.
- Reviews control charts for ICV/CCVs, LCS, CRMs, MS, and MSD. Reports excursions from warning and control limits, and monitors control limits in LIMS.
- Coordinates and conducts yearly internal audits of the entire company to ensure that appropriate quality systems are in place. Generates internal audit reports and presents them to the Operation Management group.



- Acts as project manager for all proficiency testing, intercomparison and research performance evaluation studies, and coordinate with lab and facilities for internal testing. Ensures that results are sent to required accrediting agencies in a timely manner.
- Ensures that all certification items for all accrediting agencies are maintained and in order, and that all fees are paid in a timely manner to comply with certification requirements.
- Coordinates training procedures for laboratory staff, including QA orientation and yearly ethics training.
- Ensures that all records, logbooks, SOPs, project plans, and analytical results are maintained in a retrievable fashion.
- Ensures that all documents are current as per NELAC requirements.
- Oversees facilities testing programs for reagent water, vats, bottles, equipment, and air.

The Quality Assurance Department is also responsible for the filing and archiving of all analytical data generated by Frontier GeoSciences.

4.5 Office Administration

4.5.1 Accounting Manager and Human Resource Manager

The Accounting Manager and Human Resource Manager reports directly to the president and is a member of the Operation Management group. The Accounting department is responsible for the management of financial operations, including accounting and procurement of all laboratory items. It is the Accounting Manager's responsibility to ensure that purchased items and services meet the QA Plan requirements and perform as outlined in this document.

Human resources handle the hiring of new personnel. It also administrates all personnel issues and policies. All benefits and policies are channeled from this department regarding company policy. Human resources also updates Frontier Geosciences Employee Hand Book.

4.5.2 Information Technology Services

The IT Manager reports to the Operation Manager and is a member of the Operation Management group. The IT department purchases all computer systems for laboratory operations. The IT department leads computer applications for all laboratory operations from hardware to programming applications such as: personal work stations, computers connected to analytical instrumentation, Laboratory Information Management System (LIMS), payroll software program, graphic design, and updates to Frontier Geosciences' website. The IT department also oversees electronic data deliverables (EDDs) to the specifications of the client including working with the Project Managers to ensure that



EDDs are accurate and acceptable, peer reviews EDDs before submittal to the client, and performs correction of EDDs when necessary. The IT department may deviate from written procedures per FGS-087, Deviation from Policy

This department works with the QA department to help implement QC standards through the use of electronic programming.

4.6 Safety and Facility

The Health and Safety Officer directs Frontier's safety programs to protect employees and the company from harm, to maintain safe working conditions for all, and to provide risk prevention for hazardous material exposure, accidents, fires or other unsafe conditions. In case of emergency, the Health and Safety Officer is the first point of contact. The Health and Safety Officer also develops and maintains all requirements for regulatory compliance, i.e. updating Chemical Hygiene Plan, Hazardous Waste Management Plan, Safety Evaluations, Maintain MSDS, Safety Training, etc.

Facilities management includes overseeing the optimal functioning and maintenance of the building, laboratory, office systems, electrical, mechanical, fire/life safety, common areas, and grounds. The Facilities Manager also oversees contractors for facilities projects.

4.7 Business Development

The Business Development (BD) Manager reports directly to the president and is a member of the Operation Management group. The BD Manager develops and updates the FGS website and other marketing pieces, works on different campaigns to gain new business contacts and maintain current client satisfaction of services, and looks for new tools and methods to improve customer relationship with continuous communication via various methods.

4.8 Technical Staff

4.8.1 Project Management Group

The Project Management group serves as the primary laboratory point of contact. The department monitors the progress and timeliness of analytical work and reviews ongoing work orders and all subsequent final laboratory reports for accuracy and adherence to the QA Plan. Any changes in the scope of work will be processed through this department.

4.8.1.1 Project Manager Supervisor

Reports to the Operation Manager and is a member of the Operations Management group.

The Project Manager Supervisor provides mentorship and organization to improve department efficiency, knowledge and customer service. Provides suggestions and



solutions for client interactions, dataset interpretation, report development and alternative methods.

- Acts as a working project manager to coordinate and manage client projects through all phases from inception to on-time delivery; maintains communications with clients and serves as a liaison between clients and laboratory operations to meet client needs.
- Defines project requirements to ensure all contract requirements are met and communicates requirements to appropriate personnel. Works closely with sample receiving personnel to ensure proper receipt and login of samples.
- Prioritizes client requests based on due dates and complexity of response required.
- Manages task orders, work orders, change orders and contracts for existing work.
- Writes case narratives accompanying data packages to communicate any anomalies to the client.
- Manages specialized projects that are outside of scope of lab or involve other departments.
- Creates quotes and proposals to obtain potential contracts and coordinates contract negotiations for existing contracts.
- Follows up on bids submitted and tracks status to determine if the bid is a won, lost or pending.
- Prepares LIMS invoices to clients and submits to accounting for invoicing.
- Participates in the overall sales process through direct discussions with clients and in coordination with laboratory and operations staff.
- Communicates with project managers concerning client projects, monitors workloads, provides feedback and performs employee performance evaluations.
- Reviews all major requests for proposals and assigns response to appropriate project manager.
- Trains project managers on data knowledge, providing the best analytical method to meet client demands and improving customer service responsiveness.
- Reviews all reports and quotes developed by project managers during training and periodically to ensure quality control and for staff reviews.
- Compile Project Managers forecasts for sample workload and revenue and submit to appropriate department.
- Serves as a mentor for client issues, analytical data review and report development



4.8.1.2 Project Manager

Reports to the Project Management Supervisor.

The Project Manager coordinates and manages clients' projects through all phases from inception to on-time delivery. Maintains communications with clients and serves as a liaison between clients and laboratory operations to meet client needs. Works toward achieving goals for revenue, profit and client service through the effective utilization of laboratory capacity and definition of client requirements.

- Creates work orders, change orders and sets up projects or new/existing work in LIMS with correct analytical codes and client-specific information/requests.
- Writes case narratives accompanying data packages to communicate any anomalies to the client.
- Generates and reviews final reports to ensure accuracy. Facilitates corrective action when needed.
- Generates sales orders in LIMS or QuickBooks and sends them to accounting.
- Defines project requirements to ensure all contract requirements are met and communicates requirements to appropriate personnel. Works closely with internal sample receiving personnel to ensure proper receipt and login of samples.
- Provides forecasts for projects to facilitate laboratory and financial planning.
- Demonstrates proactive commitment and adherence to industry and company safety regulations and procedures.
- Stays abreast of new developments in environmental science and chemistry applications through reading, user groups and seminars
- Manages and provides timely responses to client inquiries related to the management of projects and status of work in progress, including telephone and e-mail inquiries and requests for quotes.
- Works with laboratory and operations staff to meet client requirements and resolve service and technical issues during every phase of the project.
- Generates quotes and bid proposals to obtain potential contracts and coordinates contract negotiations for existing contracts.
- Develops business relationships with clients to further enhance client service and sales.
- Participates in the overall sales process through direct discussions with clients and in coordination with laboratory and operations staff.



- Follows up on bids submitted and tracks status to determine if the bid is a won, lost or pending.
- Prioritizes client requests based on due dates and complexity of response required.
- Follows procedures described in this Quality Assurance Plan and all applicable Frontier SOPs
- May deviate from written procedures per Frontier FGS-087, Deviation from Policy.

4.9 Mercury Supervisor

Reports to the Operation Manager and is a member of the Operations Management group.

The Mercury Supervisor for "Hg Aquatic and Other", "Emissions", and "MDN" oversees the daily operations of the entire group which include: supervision of personnel, client contact and scheduling of all projects. The supervisor oversees all tasks and provides guidance and feedback to personnel regarding performance and work product. Performs client field work as necessary.

- Manages and provides timely responses to client and Frontier personnel inquiries related to the management of projects and status of work in progress.
- Manages the MDN project including monthly data review and database updates
- Develops business relationships with clients to further enhance client service and sales.
- Performs field sampling, including sample handling, custody, processing, analysis, setup and maintenance of analyzers and related equipment.
- Performs flue gas field sampling. Monitors flue gas equipment and repairs as necessary.
- Performs data evaluation and calculation and writes reports.
- Generates quotes and proposals to obtain potential contracts.
- Reviews invoices to clients to ensure accurate entries of time and cost.
- Works with laboratory and operations staff to meet client requirements and resolve service and technical issues
- Reviews project requirements to ensure that all contract requirements met and communicates requirements to appropriate personnel.
- Demonstrates proactive commitment and adherence to industry and company safety regulations and procedures.



4.10 Hg Aquatic and Other Senior Analyst

Reports to the Mercury Supervisor and is a member of the Operations Management group.

- Oversees total and methyl mercury analysis activities for the department.
- Prepares weekly analyst schedule.
- Identifies employee development needs and takes necessary action to correct deficiencies.
- Monitors and directs analysis flow, addressing changes in priority in real-time.
- Oversees department training on mercury analyses.
- Reviews the work of the analytical staff during training periods.
- Together with Quality Assurance Officer, ensures training protocols are up-to-date and properly documented.
- Interviews incumbents along with the Mercury Supervisor to fulfill hiring needs.
- Provides input regarding employee performance, including writing and/or contributing to annual Performance Reviews and makes recommendations for promotions.
- Answers procedural or analytical questions, and provides corrective action recommendations.
- Coordinates data review
- Uses LIMS functions to track and monitor weekly analysis throughput, reruns, etc.
- Identifies maintenance needs, makes recommendations, and coordinates troubleshooting when necessary,
- Prioritizes process requests and schedules equipment to meet company priorities, with direction from operations manager, as appropriate

4.11 Emissions Project Coordinator

Reports to the Mercury Supervisor

Responsible for specialty analysis, project coordination and research project support. Will help organize and maintain sample and data flow throughout the department. Accountable for analyzing client samples, importing data, sample analysis (both routine and non-routine), sample preparation, sample disposal, technical writing, field work, and research project support.



- Performs sample analysis of total mercury, analyzes client samples, imports data into Access, and generates data tables and QA reports.
- Troubleshoots instruments as necessary.
- Provides support for sample digestion and sample disposal.
- Assists with various field sampling projects including rain water sampling or other field analysis requests.
- Provides organization, coordination and support for research projects as assigned by the department manager.
- Creates invoices for merchandise orders and sample analysis reports.
- Maintains sample tracking and data flow throughout the lab including prioritization of sample TAT, LIMS database entry, and organization of datasets and client file.
- Establishes clear follow up dates, proactively contacting others to review progress and confirm action taken.
- Ensures that development project plans adhere to the Master Project Plan throughout the project lifecycle and ensures that technical documentation is complete.
- Conducts peer review of other analysts' data.
- Trains new staff in analytical methods and data review.
- Writes/edits SOPs and other technical data.
- Demonstrates proactive commitment and adherence to industry and company safety regulations and QA procedures.

4.12 MDN (Mercury Deposition Network) Site Liaison

Reports to the Mercury Supervisor and is a member of the Operations Management group.

- The Site Liaison manages and supports the MDN site with tracking shipments, troubleshooting equipment, reading rain gauges, assisting clients with requests, and providing backup assistance with glass cleaning and preparation, shipping and receiving, sample preparation, digestion and preservation.
- Proactively tracks and manages all shipments to MDN sites, ensuring that they arrive promptly and to the correct location.
- Communicate with off-site research project staff nationwide on procedural and troubleshooting issues via phone, fax, email, and internet site.



- Assists clients with troubleshooting MDN equipment when issues arise.
- Maintains records of communications and site-related documentation.
- Gives reports and participate in discussions at oversight committee meetings twice a year.
- Maintains log books of all MDN communications and documents all conversations with clients.
- Operates a weekly precipitation sampling research site.
- Prepares and updates Standard Operating Procedures (SOPs), operation manuals, memos, QA documents, and communications related to site operations including support materials to assist site operators in operational protocols, and equipment troubleshooting and repair.
- Conducts data analysis and review from sites for completeness and accuracy, and communicate with site operators and data validation staff to ensure high quality data records are obtained.
- Troubleshoots precipitation collection and rain gauge equipment.
- Reads, interprets and monitors rain gauge reports.
- Scans weekly MOF's and charts. Follows up with the site operators to correct problems.
- Ensures that the MDN area is maintained and kept clean.
- Demonstrates proactive commitment and adherence to industry and company safety regulations and procedures applications through reading, user groups and seminars.
- Provides back up assistance with flue gas equipment and trap making.
- Together with Quality Assurance Officer, ensures training protocols are up-to-date and properly documented.
- Interviews incumbents along with the Mercury Supervisor to fulfill hiring needs.
- Provides input regarding employee performance, including writing and/or contributing to annual Performance Reviews and makes recommendations for promotions.

4.13 Trace Metal Supervisor

Reports to the Operation Manager and is a member of the Operations Management group.



- Oversees ICP-MS, AFS, Cryo, hexavalent Chromium, Iron by color analysis activities (plus various others), schedule and direct analysis workflow, and perform data review.
- Monitors/directs the performance of various preparation methods and analysis flow, addressing changes in priority in real-time.
- Conducts technical peer-review of all ICP-MS, AFS, and Cryo data including checks for completeness, calculation accuracy, data interpretation and data quality objective coherence.
- Prioritizes work, track samples, acquire data, perform calculations and statistical analysis, and store data to meet established deadlines.
- Monitors and schedules analyses of ICP-MS, AFS, Cryo, hexavalent Chromium, and Iron by color.
- Answers procedural and analytical questions and provides corrective action recommendations.
- Schedules regular instrument maintenance and service calls, coordinates troubleshooting when necessary.
- Places orders for supplies and instrument consumables.
- Assists with preparation and implementation of employee performance reviews.
- Performs and interprets required QA/QC procedures; recommends, performs and documents corrective actions. Documents possible impacts to data quality.
- Uses LIMS functions to track and monitor weekly analysis throughput, reruns, etc.
- Actively participates in the development and improvement of methods, including the writing, review and revision of applicable SOPs.
- Maintains and promotes safety and cleanliness in the laboratories in cooperation with the Health and Safety Officer. Reports health and safety concerns and problems to the Health and Safety Officer.
- Together with Quality Assurance officer, ensures that training protocols are up-to-date and properly documented.
- Demonstrates proactive commitment and adherence to industry and company safety regulations and QA procedures.
- Functions as a liaison between Analysts and Project Managers.



4.14 Mercury Analyst

Reports to the Mercury Supervisor, Hg Aquatic and Other, Senior Analyst, or MDN Site Liaison.

- Assists with the preparation and analysis of a variety of samples by various methodologies. Interprets and manages data using LIMS, Access and Excel, ensuring that data meets all established QA/QC criteria, and is made available to the Project Manager within specified time frames.
- Using established procedures, prepares, analyzes and documents sample analyses by CVAFS/EPA 1631 total mercury, CV-GC-AFS/EPA 1630 methyl mercury and other methodologies.
- Conducts review of analytical data for completeness, calculation accuracy, data interpretation and data quality objective coherence. Provides all documentation to direct supervisor for peer and scientific review of data in a timely manner.
- Troubleshoots instrument problems, performs routine maintenance, and consults with vendor technicians when appropriate.
- Manages inventory needs, assists in ordering of supplies, maintains stock and documentation of all necessary components, supplies, standards and reagents.
- Utilizes computer software to prioritize work, track samples, acquire data, perform calculations and statistical analysis, and store data.
- Prioritizes sample analysis, data management, and instrument maintenance to meet established deadlines.

4.15 Trace Metals Analyst

Reports to the Trace Metals Supervisor.

- Responsible for the preparation and analysis of a variety of sample matrices for trace metals by ICP-MS, AFS, Cryo, hexavalent chromium and iron by color analysis. The analyst interprets and manages data using LIMS, Access and Excel, ensuring that data meets all established QA/QC criteria and is made available to the Project Manager within specified time frames.
- Conducts review of analytical data including: checking for completeness, calculation accuracy, data interpretation and data quality objective coherence. Provides all documentation to group supervisor for peer and scientific review of data in a timely manner.
- Troubleshoots instrument problems, performs routine maintenance, and consults with vendor technicians (support engineers) when appropriate.



- Performs and interprets required QA/QC procedures; recommends, performs and documents corrective actions. Documents possible impacts to data quality.
- Prioritizes work, tracks samples, acquires data, performs calculations and statistical analysis, and stores data to meet established deadlines.
- Trains coworkers in sample preparation and analysis, instrument operation and maintenance, troubleshooting techniques and data quality objective adherence.
- Manages inventory needs, assists in ordering of supplies, and maintains stock of all necessary components, supplies, standards and reagents.
- Demonstrates proactive commitment and adherence to industry and company safety regulations and procedures.

4.16 Laboratory Technician

Reports to the Mercury Supervisor, Hg Aquatic and Other, Senior Analyst, MDN Site Liaison, or Trace Metals Supervisor.

Works in the laboratory assisting the Analysts, Shipping and Receiving and the Research Groups with calibrations, digestions and laboratory maintenance.

- Maintains and calibrates pipettes, including cleaning, oiling and ensuring that pipettes are in good working order.
- Prepares trace metal waters for analysis by oven digestion method and both simple and complex sample preparations.
- Prepares and fills ICP-MS tubes.
- Receives and unpacks samples, general stocking of supplies.
- Performs sampling of the facility, water and acid vats to ensure no contamination may be involved as duties.
- Ensures that the lab is maintained and kept clean.
- Assists in locating samples, or delivers samples to appropriate lab daily to improve productivity and ensure that sample hold times will not be exceeded.
- Assists with regular and ongoing sample and waste disposal.
- Wash, rinse and disinfect bottles, glassware, funnels, and thistle tubes.
- Maintains bottle inventory and container recycling.
- Assemble atmospheric mercury sample collection traps.



4.17 Shipping and Receiving Supervisor

Reports to the Operation Manager and is a member of the Operations Management group.

- Maintains and completes paperwork for all international shipments: import and export.
- Supervises the lab technicians to ensure proper tracking of shipments received, shipped and in progress.
- Provides logistical support and consultation regarding the shipment of hazardous materials and dangerous goods.
- Monitors department work product and creates and updates SOPs to reflect current methodologies.
- Oversees all laboratory inventory management for consumable items.
- Works with governmental and regulatory agencies to ensure that all shipping and receiving requirements are met; reports on problems and resolutions.
- Establishes team-building, goal-setting, good communication, motivation and acknowledgement of team success.
- Collects supply requests from all departments, usually via e-mail and create purchase orders.
- Submits purchase orders via vendor websites.
- Keeps a copy of purchase order and cross reference with shipment when it arrives and resolves any discrepancies.
- Weekly assess backorders and inform department supervisors when they can expect backorder arrivals.

4.18 Shipping and Receiving Laboratory Technician

Reports to the Shipping and Receiving Supervisor.

- Maintains shipping and receiving work area, and assists with all forms of shipping and receiving functions, including, but not limited to, Fed Ex, US Mail, UPS, shipping supplies and shipment tracking.
- Receives and unpacks samples daily, verifies accuracy of receipt against chain of custody (COC), matches it to the corresponding project and notes discrepancies.
- Logs samples into LIMS daily to assign a lot number for tracking purposes and distributes paperwork to Project Managers and Department Managers.



- Labels samples to correspond with LIMS number and places them in the proper storage area to maintain sample integrity daily.
- Notifies Project Manager and Department Managers when samples are received that have short hold times or a quick turn- a-round time so that the workload can be prioritized.
- Assists in locating samples or delivers samples to appropriate lab daily to improve productivity and ensure that sample hold times will not be exceeded.
- Performs regular and ongoing waste sample disposal.
- Performs simple sample preparation involving filtration and chemical preservation.
- Stocks, prepares and ships sample supplies to clients.
- Maintains and calibrates balances, and checks refrigerators and freezers.
- Performs bottle washing as necessary.



5 LABORATORY CAPABILITIES AND SERVICES

5.1 Laboratory Certifications

Frontier is primary NELAP certified with Florida Department of Health in the following categories: Non-potable Waters, General Chemistry and Metals, Solid and Chemical Materials-Metals and Biological Tissue-Metals.

Frontier is accredited by the Washington Department of Ecology in Air, Non-potable Water, and Solids and Chemical Materials. Frontier is also accredited by the California Department of Health, Louisiana Department of Environmental Quality, New York Department of Health and Minnesota Department of Health.

PDF files of current NELAP and state accreditation are located at:

Quality Assurance\State Accreditation\Certificates & Scopes of Accreditation.

5.2 Laboratory Experience

We have experience performing analytical chemistry, field sampling, and bench-top research to the QA specifications of the US EPA Superfund and CLP programs.

Frontier worked with the EPA to develop and codify techniques for ultra-clean sampling (US EPA Method 1669) and ultra-low level mercury analysis (US EPA Method 1631). We also developed our technique for methyl mercury in water (EPA Draft Method 1630). In addition, Frontier helped develop EPA Draft Methods 1632 (As by HG-CT-GC-AAS), 1637 (trace elements by GF-AAS with pre-concentration), 1638 (trace elements by ICP-MS), 1639 (trace elements by GF-AAS), and 1640 (trace elements by ICP-MS with pre-concentration). Frontier was selected as the EPA referee laboratory for the validation studies for all of the above EPA 1600-series methods.

5.3 Field Sampling and Training

Frontier co-developed and popularized ultra-clean sample handling protocols for environmental trace metal research and monitoring. These protocols are used to lower detection limits. In some cases, adoption of these protocols has eliminated sampling-related contamination and precluded the need for further monitoring. In 1996, Frontier helped develop and write the new EPA ultra-clean sampling protocol, Method 1669. We also implemented air-free ultra-clean sediment pore water extraction in the field. Frontier has an extensive collection of ultra-low level sampling equipment, including Teflon-coated Go-Flo bottles, peristaltic and submersible pumping systems, pre-cleaned tubing, filtration units, and thousands of Teflon bottles. We also pre-clean and modify or build low-level sampling and analytical equipment, and provide field sampling crews and on-site analyses as needed by our clients. Frontier conducts seminars and on-site training, and has authored several articles on these subjects.



5.4 Analytical Capability

Our Analytical Laboratory and Research Services include:

- Mercury Laboratory for analysis of trace level mercury in water, solids, tissues, hydrocarbons and air; monomethyl mercury in water, solids, and tissues, dimethyl mercury in water and air; and mercury in wet deposition samples.
- Mercury Deposition Network (MDN): since January 1996, Frontier GeoSciences Inc. (FGS) has served as the Mercury Analytical Laboratory (HAL) and Site Liaison Center for the MDN. The MDN, coordinated through the National Atmospheric Deposition Program (NADP), was designed with the primary objective of quantifying the wet deposition of mercury in North America to determine long-term geographic and temporal distributions. The Network has grown to incorporate over 100 sites in North America.
- Flue Gas Diagnostics, Frontier GeoSciences developed a superior method for measuring mercury speciation in flue gas and other emission sources. We test, diagnose, and recommend mercury control technology for any kind of industrial facility.
- Appendix K, Frontier GeoSciences developed the method adopted under the Clean Air Mercury Rule (CAMR) for the regulation of mercury emission from coal-fired power plants. We offer a turn-key package of equipment, service, training, analysis and reporting that is compliant with Appendix K.
- Trace metals analysis in water, solids, tissues, and hydrocarbons by inductively-coupled plasma mass spectrometry (ICP-MS) equipped with or without a Dynamic Reaction Cell (DRC).
- Se and As analysis in water, solids, and tissues as well as speciation by hydride generation - atomic fluorescence spectrometry (HG-AFS).
- Parts per trillion As speciation in waters and tissues by hydride generation cryogenic trapping gas chromatography atomic absorption spectrophotometry (HG-CT-GC-AAS).
- As, Se, and Cr, speciation analysis by inductively-coupled plasma mass spectrometry operating with a Dynamic Reaction Cell (ICP-MS-DRC) equipped with a High-performance liquid chromatography (HPLC) system.



6 QUALITY ASSURANCE COMPLIANCE and OBJECTIVES

Frontier's data includes all of the NELAC requirements regarding the following:

- Sample tracking and preservation
- Traceability of standards and reagents
- Proficiency samples. Samples of double blind standards used to check analytical procedures for accuracy and precision.
- Control Charts for ICV, CCVs, LCS/LCSDs, and MS/MSDs. Used to monitor precision and accuracy in all analytical test methods. A statistical evaluation from our database to set control limits for individual parameters.
- Use of current SOPs (SOPs are reviewed annually)
- Use of performance based measurement systems when possible
- Determination of practical quantitation limits (PQL) for all analytes in every test method we perform
- Training records of analysts

Definitions of QC terms and requirements.

Precision is a measure of our ability to use our methods to analyze a sample repeatedly and get the same results each time. To demonstrate the precision of a method, sample duplicates are analyzed and their results compared.

Accuracy or bias, is a measure of how close the result is to the true or expected value of target analyte in the sample. Accuracy may be determined by the analysis of standard reference materials, blank spikes and matrix spikes, where the result can be compared with a true or expected value.

Representativeness describes how well a single sample can characterize the conditions of the entire sample population. Appropriate sampling techniques and artifact free procedures, combined with sample homogenization, help to achieve representative data.

Comparability is a particularly important QA criterion for ongoing projects. Individual data sets are evaluated with respect to other data from that project to ensure validity and scientific coherence.

Completeness is a measure of how many data points collected are usable; Frontier considers 95% of usable data to be an acceptable value for completeness.

Frontier provides data packages in one of two Quality Assurance formats, "Standard-Level" and "High-Level". The two different levels do not represent differences in



analytical quality. Rather, the differences are in the degree of documentation, and therefore the ability to defend the data in legal proceedings. The quality of data produced under the two QA reporting schemes, as measured by quantitative indicators such as precision, accuracy (bias), and detection limits, are equivalent.

In addition, Frontier will provide custom QA/QC packages to meet specific project needs.

6.1 Proficiency Testing Program

As part of good laboratory practices, Frontier participates in proficiency test studies at least six times per year. We currently participate in semi-annual New York Department of Health performance studies for non-potable water/solid & chemical material/ air & emissions chemistry proficiency test and we also participate in two water and two soils pollution proficiency tests each year. These are supplied by a licensed and approved commercial provider. Results for each of these studies are submitted to all of Frontier's certifying officers, and are available to any client upon request.

6.2 Laboratory Inter-comparison Studies

Each year, Frontier takes part in several laboratory inter-comparison studies, spanning a wide range of matrices including biota, sediment, estuarine waters, and fresh waters. Frontier is a regular participant in studies prepared by the United States Geological Survey (USGS), the National Research Council of Canada (NRCC), the International Atomic Energy Agency of Monaco (IAEA), and the National Water Research Institute (Canada). Typically, our larger projects specifically include additional inter-comparison studies.

Major laboratory comparison exercises conducted by Frontier include the CALFED Mercury Project Inter-comparison Studies and EPRI-sponsored International Mercury Speciation Inter-Comparison Exercise (Bloom, et al, Wat. Air Soil Pollut. 1995). Frontier has also participated in the certification of International Atomic Energy Agency (IAEA), National Research Council of Canada (NRCC), and National Institute of Standards and Technology (NIST) reference materials for mercury, methyl mercury, and other trace elements. We also participated in the Mace Head, Ireland, international atmospheric mercury intercomparison and Steamboat Springs (NV) soil Hg flux intercomparison (Ebinghaus, et al, Atmos. Environ. 1999). Frontier served under contract to the US EPA as one of the beta testing labs for Method 1632 (low level arsenic by HG-CT-GC-AAS). We have also provided external laboratory comparisons for numerous other laboratories and projects, setting up or participating in approximately six inter-comparisons and/or standards checks per year.

6.3 Training Program

Staff members are trained in new skills or methods by a mentorship process. New staff members (or staff members learning a new skill or method) are assigned to their immediate supervisor or a senior coworker. The new method is taught according to the following steps:



- Reading the SOP.
- Observing performance of the method.
- Closer reading of the SOP, associated literature, and other notes.
- Supervised practice of the method on non critical work until the supervisor is satisfied that the employee is competent.
- Unsupervised practice of the method, with review by the trainer and supervisor.
- Unsupervised performance of the method and completed Demonstration of Capability (DOC).
- Completion of these steps is documented on a training form, which is signed by the trainee and the applicable supervisor. Employee training files are reviewed during internal audits.



7 SAMPLE HANDLING

7.1 Sampling Procedures

All samples for trace metals analysis must be collected in a manner that neither contaminates, loses, nor changes the chemical form of the analytes of interest. The appropriate sampling technique may vary depending on the location, sample type, sampling objective, client sampling plan, etc.

Frontier SOP FGS-008, "Ultra-Clean Aqueous Sample Collection" (Modified EPA Method 1669), should be referenced for sampling water matrices. Sediment, air, and tissue sampling protocols are project dependent.

The use of appropriate sampling equipment and sample containers is an integral part of the sampling method. Frontier maintains a stock of sample bottles of the preferred types (Teflon, glass, HDPE, polycarbonate) for ambient water sampling. Frontier cleans, tests, and bags these bottles according to procedures described in Frontier SOPs FGS-007 "Cleaning of Sampling Equipment and Bottles for Mercury Analysis" and FGS-065 "Cleaning of Sampling Equipment and Bottles for Analysis of Trace Metals." Frontier routinely supplies these bottles to clients to facilitate sampling programs.

Frontier staff is experienced at selecting appropriate sampling techniques on the basis of data quality objectives and project requirements. The Frontier Project Manager should be contacted for direct consultation regarding sampling plans.

Sub-sampling in the laboratory must be done following protocols in Frontier SOP FGS-078, "Sub-Sampling." This procedure aims to ensure thorough sample homogenization. Samples that are not properly homogenized may lead to irreproducible results.

7.2 Sample Acceptance

It is the client's responsibility to provide a chain-of-custody (COC) form with submitted samples. If no COC is provided, the Project Manager will be notified, and the Project Manager will attempt to contact the client (unless otherwise directed by the Project Manager). If the client is reached, but is unable to fax a copy of the COC, or if no COC has been generated, Frontier's Shipping & Receiving Technician or the Project Manager will create one. If the client cannot be reached, the Project Manager, Project Manager Supervisor or the Operations Manager will decide whether to assume responsibility for the shipment.

Clients have the responsibility of ensuring that samples are shipped with consideration to preservation (appropriate chemical or cold preserved) and time sensitivity. Proper containers must be used when collecting samples. Frontier depends upon clients to ensure that proper attention is given to packing and shipping sample containers. Finally, proper attention must be given to the packing materials used in sample shipment.



Note: Samples may not be sent packed in vermiculite, as the dust from this material represents a contamination and human health risk. The client should use bubble wrap or foam as packing materials.

The Project Manager, working in communication with the client, will decide how best to proceed with sample receipt in any of the following events.

- Samples arrive at Frontier improperly preserved or outside of pre-established time parameters.
- Inappropriate sample containers are used for sample collection.
- Sample identification discrepancies.
- Sample containers break or leak during transport to Frontier.
- Inappropriate packing materials are used for shipping samples to Frontier.

Samples that are potentially radioactive must be pre-approved by the Radiation Safety Officer (Safety and Facilities Manager) before the samples are sent to Frontier GeoSciences. All potentially radioactive incoming samples must be accompanied by client-supplied certification containing: radionuclide(s) and activity (mCi). Potentially radioactive samples shipments that arrive without client-supplied certification will not be received. Potentially radioactive samples must be handled, following SOP FGS 030, "Handling of Potentially Radioactive Samples."

Frontier will not accept hazardous samples without prior agreement that the client is responsible for sample handling and disposal after the analytical report is provided. Frontier reserves the right to reject any samples that may pose a reasonable threat to the health or safety of personnel (e.g., unsterilized human biological tissue, radioactive materials, unknown industrial wastes, etc.). If, upon opening a package, the contents are unacceptable because of safety concerns, the package(s) will be resealed, placed in a secure outside storage area, and the client will be notified.

Soil samples must be accompanied with appropriate documentation according to Frontier SOP FGS-135, "Soil Permit SOP for Compliance with the USDA, APHIS Soil Permit Compliance Agreement" This SOP is designated to apply to all soil samples received at Frontier GeoSciences, Inc. There will be no delineation or separation of the soil origins.

Should the client not include their soil permit with the shipment, Shipping and Receiving will notify the Project Manager. The Project Manager will immediately contact the client to obtain the permit



7.3 Sample Receipt

Samples are delivered to the laboratory sample receiving area by private or public mail carrier, courier, or project personnel. Any staff employee may sign the sample receipt form. The Shipping & Receiving Technician breaks the custody seal (if present), opens the container, and measures the temperature. The COC is located and the contents are unpacked. The general condition of the samples is noted (e.g., broken or leaking containers, unusual temperature) and samples are compared to the COC for discrepancies. Once the COC is verified it is signed, scanned and saved into LIMS "Laboratory Information Management System." The received samples are given a unique work order identification number (i.e. 0806001, year 08, month 06, first work order to be received during the month of June) when logged into LIMS.

Once the samples are properly identified, all sample receiving information is entered into LIMS which includes but are not limited to:

- Client name
- Project name
- Analysis requested
- Lab project manager
- Date and time received
- Date and time logged in
- Due date,
- TAT (turn- around- time)
- Received by
- Logged in by
- Shipped by
- Tracking number
- Shipping containers(numbers of coolers and receiving temperature)
- Condition of the coolers (custody seal, Container intact, COC/labels agree, preservation confirmed, received on ice)

The Project Manager will use the work order comments for specific sample requirements and the Shipping and Receiving Technician will use the MMO section to note any sample discrepancies.



The Shipping & Receiving Technician will then notify the Project Manager of the shipment and transfer the samples to the preparation laboratory location.

It is the responsibility of the client to notify the laboratory if the samples are potentially very high in metals. If there is a shipment that may contain samples with very high levels of trace metals (e.g., from a contaminated site), the Shipping & Receiving Technician is required to notify the Project Manager before opening the shipping container. If the sample shipment is identified as potentially high in metals (particularly in mercury, which can have a significant gaseous component), a screening procedure by the LUMEX real time low level atmospheric Hg detector is performed to determine whether the shipment can be safely stored in the laboratory.

Each project is assigned to a specific Project Manager. The Project Manager is ultimately responsible for sample receipt, chain of custody, tracking, integrity, preservation, transport, storage, analysis, data validation and disposal. If the Project Manager (or his/her backup) is unavailable for decision-making at any point from sample arrival to sample disposal, the Project Manager Supervisor, the Trace Metals or Mercury Supervisor will be notified, and will take responsibility until the Project Manager returns.

7.4 Chain of Custody

Once the COC is verified and signed by Shipping & Receiving Technician sufficient copies are made and the COC gets scanned into LIMS. The original is given to the Project Manager, and copies following the work order are distributed to the sample storage area. If there are separate bottles for mercury analysis and other trace metals analysis, a copy of the COC form is included with both sets of samples to each laboratory. Clients may also request an internal tracking COC form.

A sample is considered to be "in custody" if it meets the following criteria:

- (a) It is actually in the Project Manager's, Analyst's or Laboratory Technician's possession.
- (b) It remains in the Project Manager's or Analyst's visual range once possession of the sample has been assumed, or
- (c) The sample has been stored in a secure area.

To satisfy these custody provisions, the laboratory follows the following procedures:

- (a) Samples are stored in a secure area,
- (b) Laboratory doors are locked at all times,
- (c) Non-frequent visitors are always accompanied by a member of the laboratory staff, and



(d) Samples remain in the secure area until the Project Manager has given permission for their disposal or until they are returned to the client.

7.5 Sample Tracking

The documentation for tracking samples from receipt through disposal includes all COC records (including internal tracking COC when applicable), sample receipt logbooks, laboratory bench-sheets, laboratory notebooks, instrument printouts (raw data), and final analytical reports. The Project Managers, Laboratory Technicians, Analysts, and Shipping & Receiving Technicians are in constant communication. Because the laboratory is small and secured, samples are stored in secure refrigerators only if they are awaiting speciation analysis, or at the request of the client. After analysis, samples are placed in the sample storage container. When the Frontier designated holding time has elapsed, samples are disposed of (or returned to the client). The current location of a sample can always be found in LIMS under work order/samples/location.

7.6 Tracking Specifics for the Mercury Group

When analyzing for both total mercury and methyl mercury, the samples are preserved to 0.4% HCl and stored in a secure refrigerator. Following distillation of samples for methyl mercury, a small sticker or tape labeled with the date of distillation is placed on the lid of each bottle. Once the methyl mercury data has been cleared, samples are transferred to the sample receipt counter in the Mercury Laboratory, and are marked for total mercury preservation. Alternatively, a split for total mercury analysis can be taken 24 hours after initial preservation and after pH verification. Aqueous samples are preserved with bromine monochloride (BrCl) at least 24 hours before analysis. All solid samples are logged into an appropriate freezer. After analysis, aqueous samples not requiring refrigeration are placed on shelves in the Mercury Laboratory. As time allows, samples are logged into the secure sample storage container for long-term storage. Solid and aqueous samples requiring refrigeration are returned to their original locations until disposal.

7.7 Tracking Specifics for the Trace Metals Group

After samples are preserved and checked for pH, they are stored in the Trace Metals Laboratory at room temperature. Samples for speciation are checked for pH and stored in a secure refrigerator. Water samples awaiting a digestion, extraction, or other preparative step remain in the Trace Metals Laboratory. Solid samples awaiting a digestion, extraction, or other preparative step are logged into a secure refrigerator or freezer. When samples are ready for analysis, they are stored in the appropriate analytical laboratory. When all analyses are complete, the samples are stored in a cupboard or in the secure sample storage container until permission for disposal is granted.

7.8 Mercury-Specific Sample Handling

For low-level (ambient) mercury analyses in water samples, only acid-cleaned Teflon containers (or borosilicate glass or quartz containers with Teflon-lined lids) may be



used. Tissues, sediments, and contaminated water samples should be stored in cleaned, glass containers with Teflon-lined lids. Frontier cannot take responsibility for potential sample contamination resulting from the use of polyethylene, polypropylene, or other plastics not approved for mercury work. Alternate containers may be used if documented testing shows that the container type does not contaminate or absorb mercury in solution.

7.9 Trace Metals Specific Sample Handling

For low-level (ambient) trace metals analyses of samples preserved to 1% Nitric acid new engraved polyethylene bottles cleaned by rinsing 3 times with reagent water and placed in a class-100 clean clean-air station until dry according to SOP FGS-065 "Cleaning of Sampling Equipment and Bottles for Trace Metals Analysis" are preferably used. Polycarbonate, Teflon, or glass containers (with Teflon-lined lids) may be used. Please note that glass containers are inappropriate for some analytes such as boron, potassium, silicon, and low-level metals. If clients submit samples in their own bottles, bottle blanks should also be submitted.

7.10 Shipping of sample

For both mercury and trace metals analyses, aqueous samples should be sent by overnight courier. Samples submitted for speciation analysis should be cold preserved. Solid samples should be preserved by freezing in the field, unless requested otherwise. Each sample container should be sealed inside a zip-type bag and labeled with a unique sample number. After the Shipping & Receiving Technician has relinquished custody to the Project Manager, the samples are stored in the refrigerator, freezer, or shelf space designed and allocated for sample storage. All company employees have access to the sample storage area, which is within the secure analytical laboratory. After samples are used, they are taken to the sample storage container to be held for a designated holding time before disposal.

7.11 Disposal

Samples must eventually be disposed of to preserve laboratory storage space. Timely and proper disposal is emphasized for the sake of efficiency, and, in the case of hazardous substances, for safety. All samples are held for at least one month after the completion of a project. After that time they are disposed of, or returned to the client. Clients may request a longer holding time in writing from the Project Manager.



8 TRACEABILITY

8.1 Documentation

Frontier's goal is to be able to trace all laboratory measurements to their sources. The laboratory uses traceable reagents, standards, and reference materials in all procedures. Furthermore, all standard solutions and analytical reagents are tested for suitability before use. Testing is documented, and the documentation is kept in the QA Office or with the applicable group supervisor. Instrument calibration, reagent and bottle testing, and equipment maintenance are all thoroughly documented.

All calibrations are traceable to certified standards or manufacturer lot numbers. For analytical instruments, high purity calibration standards are obtained from chemical suppliers. Certificates attesting to the concentration ranges of the covered analytes are retained in the QA Office

8.2 Reagents and Standards

Standards and reagents are documented in LIMS upon receipt or creation. A LIMS generated label is affixed to each standard and reagent, with the name of the solution, the person who prepared or received it, the date it was prepared or received, and the expiration date.

For all standards, LIMS documentation must include the following: a description of the standard, department, expiration date of the standard (not to exceed the expiration of the parent standard), the name of the person that made the standard or reagent, the date it was prepared (or received), number of containers, final volume, a reference date (date entered into LIMS), concentration units ($\mu\text{g/mL}$), the vendor and the vendor lot. The solvent lot is not applicable. In the comments section, the analyst must enter the work order number for traceability purpose. The correct parent standard must be noted, as well as the aliquot used. Analytes are entered individually. From the list, LIMS will calculate the true value of the standard based on the aliquot of the parent and the final volume. Click the appropriate radio button under "Standard Type". A Spike Mix is a standard that is used in the bench sheet, and a Calibration standard is a standard used in sequences. A Reference Standard is a Certified Reference Material (CRM). The analyst must click the standard to "Inactive" until approved by QA.

Procedures for standards documentation are detailed in SOPs FGS-074, "Stock and Working Standards for Trace Metals Analysis," FGS-069, "Total Mercury Analysis by Cold Vapor – Atomic Fluorescence Spectrometry (CV-AFS)," and FGS-070, "Methyl Mercury Working Standards and Instrument Calibration."

Stock standards and CRMs are logged into LIMS upon receipt by Shipping and Receiving (S&R) and later verified by the Quality Assurance department (QA). Where possible, Frontier uses reference materials that are certified and traceable to national or international standards of measurement. These do not require testing, provided there is a Certificate of Analysis on file in the QA office. When receiving a solid CRM, QA



generates a work order in LIMS for total solids analysis. The QA Office maintains a file of the original certificates of analysis in a three-ring binder.

Neat reagents are logged into LIMS upon receipt by the Shipping and Receiving Department. Reagent identifiers are assigned based on lot number. When a new lot number is received from the vendor, a new unique identifier is created for that lot number. Reagents from an existing lot are given the same identifier as the rest of that lot.

Working standards are prepared by the analyst, logged into LIMS and assigned a unique identifier. For calibration standards, LIMS documentation will include one entry reflecting the amount of parent standard used per final volume, and several entries for the calibration points at the instrument (SEQ-CAL1, SEQ-CAL2, etc) reflecting the amount of standard and default aliquot at the instrument. For the ICV standard, LIMS documentation will include one entry reflecting the amount of parent standard used per final volume, and one entry reflecting the amount of standard used and default aliquot at the instrument. On the preparation date, the analyst notifies QA that a new standard has been prepared and requests a work order to document standard testing. Once the work order is generated, the analyst enters the work order ID into the comments section of the standard.

Working reagents are prepared by the analyst, logged into LIMS and assigned a unique identifier. Reagents entered into LIMS must have the information listed in section above with the following exceptions: the parent neat reagents are added by their unique identifier, but the amount of each reagent does not need to be entered. It is not necessary to enter analytes from the list for reagents. The Solvent Lot is not applicable to working reagents. The radio button must be clicked to Reagent. If the reagent requires testing, QA will need to be notified, so they can create a work order prior to testing. All reagents used during analysis and prep should be added to the bench sheet.

Additionally, reagents undergo continuous monitoring through analysis of method blanks. A method blank is a sample of reagent water and analytical reagents that goes through the same analytical process as the corresponding samples. A minimum of three method blank samples are prepared with each analytical batch.

The performance of analytical support equipment such as balances and refrigerators is checked daily, pipettes are checked weekly, and pH meters and conductivity meters are checked before use. In addition, all analytical support equipment (balances, thermometers, ovens, pH and conductivity) is calibrated or verified by a certified metrology laboratory no less than once a year. When support equipment is checked or calibrated, measurements are recorded in laboratory logbooks or in the calibration file in the QA Office.



9 TEST METHODS AND STANDARD OPERATING PROCEDURES

9.1 Test Methods and Standard Operating Procedures (SOPs)

Frontier utilizes state-of-the-art analytical techniques. Some are in use by the scientific research community, and some are under development at our own research lab and not yet available as promulgated methods. Listed below is a brief description of our most commonly used analytical methods, as well as the equivalent EPA or Standard Methods reference.

Frontier's methods are formally reviewed each year, and are periodically updated to represent the latest analytical knowledge of the research community, and/or to improve efficiency. The QA Officer and Operations Manager are responsible for approving new and revised SOPs prior to implementation in the laboratory. The QA Officer ensures that all Frontier staff members are provided with the most recent copy of each relevant SOP. All SOPs are also given a sequential Frontier number, followed by the revision number. For example, SOP number 1, revision 3, would be labeled as FGS-001.3. This identification is printed on each page of every SOP.

All SOPs are considered and treated as proprietary information, protected by the Washington State Trade Secret Act, RCW 19.108 et seq., and other laws. Proprietary information shall be kept in the strictest confidence and shall not be used or appropriated for the benefit of any party without the prior written consent of Frontier.

Frontier Standard Operating Procedures

FGS-002	Balance Calibration and Maintenance
FGS-003	Pipette Calibration and Maintenance
FGS-004	Refrigerator and Freezer Calibration and Maintenance
FGS-005	Sample Receipt, Chain of Custody, Tracking, and Disposal
FGS-007	Cleaning of Sampling Equipment and Bottles for Mercury Analysis
FGS-008	Ultra Clean Aqueous Sample Collection
FGS-009	Digestion for Gas/Air Samples Collected on Iodated Carbon Traps for Total Mercury Analysis
FGS-010	KOH/Methanol Digestion of Tissues for Methyl Mercury Analysis
FGS-011 (70:30)	Digestion of Tissues for Total Mercury Using Nitric and Sulfuric Acids
FGS-012	Oxidation of Aqueous Samples for a Total Mercury Analysis



FGS-013	Distillation of Aqueous Samples for Methyl Mercury Analysis
FGS-016	Total Suspended Solids in Aqueous Samples
FGS-017	Distillation of Low Level Solids for Methyl Mercury Analysis
FGS-019	Total Solids and Loss on Ignition Determination for Tissues and Sediments
FGS-022	Determination of Arsenic Species in Water by HG-CT-GC-AAS
FGS-029	Ultra-Clean Sample Filtration
FGS-030	The Handling of Potentially Radioactive Samples from Oak Ridge National Laboratory
FGS-031	Mercury Digest for Gas/Air Samples Collected on KCl/Quartz or KCl/Lime Trap
FGS-032	Extraction of Ag, Cd, Cu, Pb, and Ni from Water by Co-APDC
FGS-038	Data Review and Validation
FGS-039	Corrective Action File
FGS-040	Document Management System
FGS-041	Internal Quality Assurance Audit
FGS-043	Data Correction
FGS-045	Preparation of Sediments for Determination of Methyl Mercury in Sediments by Acidic KBr Extraction into Methylene Chloride
FGS-046	Preparation for Leachable Inorganic Selenium Speciation in Sediment and Soil Samples by Selective Leaching (research)
FGS-047	Preparation for Leachable Inorganic Selenium Speciation in Tissue Samples by Selective Leaching (research)
FGS-048	Creation and Control of SOPs
FGS-049	Dissolved Iron Speciation in Aqueous Samples by Colorimetric Detection
FGS-050	Sample Receipt, Handling, and Disposal of Samples Potentially Containing Hazardous Materials
FGS-051	Total Mercury Extraction from Liquid Hydrocarbons (research)
FGS-052	Total Recoverable Metals Digestion by Oven Heating



FGS-054	Determination of Trace Elements by Inductively Coupled Plasma - Mass Spectrometry Using a PE Elan 6000
FGS-055	Determination of Selenium Species in Water by Hydride Generation - Atomic Fluorescence Spectrometry
FGS-056	Washing Feather and Hair Samples Prior to Trace Metals Analysis
FGS-058	Total Metals Digestion for Animal or Plant Tissues
FGS-059	Determination of Total Reducible Arsenic in Water by Hydride Generation - Atomic Fluorescence Spectrometry
FGS-060	Determination of Hexavalent Chromium in Waters by Colorimetric Detection
FGS-061	Gold Trap Construction
FGS-062	Preparation of Carbo-Traps for Methyl Mercury Analysis
FGS-064	Leaching of Inorganic Arsenic Species from Tissue Samples
FGS-065	Cleaning of Sampling Equipment and Bottles for Analysis of Trace Metals
FGS-066	Preparation of Solids Samples for Total Mercury Analysis by Modified Cold Aqua-Regia Digestion
FGS-069	Total Mercury Analysis by Cold Vapor Atomic Fluorescence Spectroscopy (CVAFS).
FGS-070	Methyl Mercury Calibration and Analysis
FGS-071	Extraction for Leachable Arsenate from Soils and Sediments (research)
FGS-072	Ordering Supplies
FGS-073	Sample Storage Container
FGS-074	Stock and Working Standards for Trace Metals Analysis
FGS-078	Sample Preservation and Sub-Sampling
FGS-080	Generation of Electronic Data Deliverables
FGS-081	Document Management for Raw Data
FGS-084	Total Recoverable Metals in Sediments and Soils via a Modified Aqua Regia Oven Bomb Digestion
FGS-086	Equipment Maintenance Records



FGS-087	Procedures for Deviation from Laboratory Policy
FGS-088	Leachable Arsenite Extraction from Soils and Sediments (research)
FGS-089	Sputter Coating Quartz Sand
FGS-090	Selective Sequential Extraction of Geological Samples for the Determination of Biogeochemically Relevant Inorganic Mercury Fractionation (research)
FGS-091	Determination of Total Reducible Antimony in Water by Hydride Generation - Atomic Fluorescence Spectrometry
FGS-092	Software Documentation, Data Security, and Backups
FGS-094	Documentation of Personal Training
FGS-095	Contract Review
FGS-096	Acid Vat Monitoring Program
FGS-097	Customer Complaint Procedures and Files
FGS-098	Dimethyl Hg in Environmental Media (research)
FGS-099	Waste Dumping Procedure for Client Sample Waste
FGS-101	Traceability Protocols
FGS-104	Generation of Hardcopy Sample Result Reports
FGS-105	Control Charting
FGS-108	Extraction of Hexavalent Chromium from Tissue Samples
FGS-109	Reductive Precipitation
FGS-110	Digestion of Bayer Process Liquor for Mercury and Trace Metal Analysis
FGS-111	HF/Nitric/HCl Digest for Mercury followed by Nitric Evaporation for Trace Metals
FGS-113	Headspace Analysis for Mercury (research)
FGS-114	Selective Sequential Extraction of Solid and Sediments for the Determination of Inorganic Arsenic Fraction
FGS-116	Total Dissolved Solids (TDS)
FGS-118	EPA 1638 Modified (2006)



FGS-119	EPA 200.8
FGS-120	HOT BLOCK DIGESTION –EPA 200.8
FGS-122	Total Metals and Mercury Digestion for Various Matrices (Hydrocarbon, Surfactant, Tissue) Using the Anton-Paar High Pressure Asher (HPA-S)
FGS-136	Analysis of Air Hg Emissions Via The Solid Sorbent Method
FGS-137	Determination of Total Mercury in Various Matrices by Cold Vapor Atomic Fluorescence Spectrometry (EPA Method 1631E)

EPA Method 200.8: Determination of Trace Elements in Drinking Waters and Wastewaters by Inductively Coupled Plasma-Mass Spectrometry

Draft EPA Method 1630 Modified: Methyl Mercury in Water by Distillation, Aqueous Ethylation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry

EPA Method 1631 Modified: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry

EPA Method 1638 Modified: Determination of Trace Elements in Ambient Waters by Inductively Coupled Plasma-Mass Spectrometry

Standard Method 2340 B: Hardness

Standard Method 3500 Cr-D: Colorimetric Method



10 INTERNAL QUALITY CONTROL

10.1 Laboratory Quality Control Samples

The laboratory uses control (QC) samples to assess the validity of analytical results. QC samples include instrument blanks, preparation blanks, initial and continuing calibration verification standards, initial and continuing calibration blanks, reference materials, duplicates, and spiked samples. QC samples are analyzed in the same way as field samples, at a frequency described either in this QAP, the applicable SOP, or in the contract with the client. If the QC sample results fall within the acceptance criteria (also detailed in the method, this QAP, or contracted by the client), the analytical data is valid and acceptable.

Of particular importance to the client is Frontier's position that a single non-compliant result on a QC sample does not automatically invalidate a data set. All non-compliant results are investigated, corrective actions will be generated if needed, and the issue is communicated to the client in the report narrative. Conversely, the Quality Assurance Officer may invalidate data for reasons of scientific coherence, even if all QC parameters are within acceptable limits.

Non-compliant results on QC samples from field work do not necessarily reflect analytical problems. Unless samples are collected by Frontier staff, Frontier will not be responsible for poor results on field samples if laboratory QC results are acceptable. Additionally, field QC sample data that are non-compliant are not flagged or interpreted by Frontier. For analytical questions regarding data review consult "AL Group Data Reviewer Handbook" and follow Qualifier Flowcharts located at:

Quality Assurance\Data Review\Data Review Reference\Data Review Flowcharts

10.2 Calibration of Analytical Instruments

Every instrument used to analyze samples at Frontier must pass the calibration criteria in the relevant SOP. Initial calibration criteria for instrument reproducibility and sensitivity must be met before samples may be analyzed. Continuing calibration verification (CCV) checks establish whether ongoing instrument calibration is acceptable.

Due to the variety of methods and instruments used at Frontier, individual SOPs must be referenced for specific calibration protocols. In general for Trace Metals SOP FGS-054 "Determination of Trace Elements by Inductively Coupled Plasma - Mass Spectrometry (PBMS)" the Calibration is based on a five-point calibration, forced through the origin, with a correlation coefficient (R) greater than or equal to (\geq) 0.995.

For total mercury SOP FGS-069.4.1 "Determination of Total Mercury in Various Matrices by Cold Vapor Atomic Fluorescence Spectrometry (Modified EPA Method 1631E)" and methyl mercury FGS-070.3 "Determination of Methyl Mercury in Various Matrices by Cold Vapor - Gas Chromatography - Atomic Fluorescence Spectrometry (CV-GC-AFS)"



calibrations start with a five point calibration curve with a Calibration Factor RSD less than or equal to (\leq) 15 %.

10.3 Initial and Continuing Calibration Verification Standards (ICVs and CCVs)

An ICV is analyzed following each calibration curve to verify the accuracy of the primary standard solution. The ICV is a solution made from a second source standard, independent of that used in the primary standard solution. CCVs verify that the analytical system is in control, or demonstrates analytical drift. The CCV is a standard solution that is made from a traceable stock standard (usually the same source as the primary calibration stock). CCVs are analyzed at a frequency of every ten samples or less and at the end of each analytical sequence. All ICV/CCVs references a unique identification number and are traceable through LIMS. All raw data references a unique laboratory ID number and includes a unique identifier for each standard used in the analysis. These identification numbers are traceable through LIMS.

10.4 Initial and Continuing Calibration Blanks (ICBs and CCBs)

Instrument blanks are used to demonstrate freedom from system contamination, carryover, and to monitor baseline drift. An ICB or CCB is analyzed immediately following the ICV or CCV, respectively.

10.5 Method Blanks

A method blank is a sample that contains only analytical reagents, yet undergoes the same analytical processes as the corresponding client samples. Method blanks are used to monitor laboratory performance, and to detect contamination that could have been introduced during the analytical procedure. At a minimum, three method blanks are required per batch.

Method Blank (BLK) or Preparation Blank (PB), for waters, an aliquot of reagent water that is prepared and analyzed in a manner identical to that of samples. For digested solids, method blanks consist of the same reagents used to digest the samples, in the same volume or proportion and are carried through the complete sample preparation and analytical procedure. Boiling chips are used as a blank matrix for solids.

10.6 Certified Reference Materials (CRMs)

Reference materials are matrix specific standards that are accompanied by a certificate of analysis. Frontier generally purchases reference materials from the National Institute of Standards and Technology (NIST), the National Research Council of Canada (NRCC), or the International Atomic Energy Agency (IAEA). Frontier maintains the position that matrix equivalent reference materials are the best measure of precision and accuracy (bias), as issues associated with matrix type and homogeneity may be assessed. Unfortunately, reference materials do not exist for all matrices or analytes. Frontier will utilize reference materials with each analytical run whenever the appropriate matrix is available.



10.7 Matrix Duplicate/Triplicate (MD/MT)

Replicate samples provide information about analytical precision. Frontier generally analyzes one matrix duplicate (MD) of a matrix representative sample per each batch of twenty samples. The relative percent difference (RPD) between these two replicates should be less than 20%, or according to method. When a sample is analyzed in triplicates (MT) the relative standard deviation (RSD) between the samples is calculated and should be less than 20%, or according to method. If an MD/MT fails, and the sample concentration is greater than 10 times the PQL, the ambient sample and the duplicate should be reanalyzed for confirmation, if possible. The undigested sample should be inspected for heterogeneity, and a note made in the work order (MMO) and/or data review checklist.

For samples that cannot be homogenized, such as Hg⁰ contaminated soils, splits will be considered as functionally equivalent to field replicates. The frequency of replicate analysis is specified in the client's quality assurance project plan or contract. The laboratory has no control over field and sampling induced variability, and so the relative precision of field duplicates is viewed as serving informational purposes only.

10.8 Matrix Spikes/Matrix Spike Duplicate (MS/MSD), Analytical Spike/Analytical Spike Duplicate (AS/ASD)

The purpose of analyzing matrix spikes and matrix spike duplicates (MS/MSD) is to demonstrate the performance of the analytical method in a particular sample matrix, and to recognize matrix interference. In this type of analysis, predetermined quantities of the analyte are added to a sample matrix before (when possible) sample extraction or digestion. If the sample is spiked with the analyte of interest after extraction or digestion this is considered an analytical spike and an analytical spike duplicate (AS/ASD). If an MS/MSD fails, an AS/ASD should be analyzed, if possible. The purpose of the AS/ASD is to ascertain that the largest aliquot of a sample can be analyzed at without matrix interference. The AS/ASD should be spiked 1 to 5 times the ambient concentration.

10.9 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)

A Laboratory blank spike is a sample of reagent water or analytical reagents that has predetermined quantities of analyte added. It undergoes the same preparation and analytical processes as the corresponding samples. Blank spikes are used to evaluate the daily performance of a method, but are not subject to matrix effects that may occur in matrix spikes. They are used primarily when no appropriate reference material is available for a particular matrix.

10.10 Method Detection Limits

The Method Detection Limits (MDL) are determined according to 40 CFR Part 136, Section B. Ten replicates (9 degree of freedom) are run with a sample that is spiked 3-10 times the expected MDL. The standard deviation (σ) taken from the resulting data



and the MDL is calculated as follows: $MDL = 2.821 * \sigma$. This value should not be interpreted as the method reporting limit.

The Practical Quantitation Limit (PQL) is the reporting limit for the method and the control limit of the method for the specified analyte (2003 NELAC regulation 5.5.5.2.2.1.h.3). The PQL is determined by running ten replicate samples with a concentration that will produce a recovery of 70-130%. When possible the same samples used for the MDL study are used to generate this data. The PQL is referred to as the Method Reporting Limit (MRL) in LIMS. The PQL is then incorporated as the lowest standard of the calibration curve, where the standard recovery must be within the same predetermined control limits as the initial study.

MDL and PQL data is found in the QA Office and the LIMS. Also, current MDL and PQL studies can be found on the server by going to: Quality Assurance\MDLs PQLs & RLS\Complete Reports of MDL Studies by Analyte and Matrix\Current

10.11 Method Validation

No analytical work can be performed on client samples until the Operations Manager and Quality Assurance Officer have officially approved the method. An exception is made if a method is being developed or researched in consultation with the client. All method validation documentation is stored in the QA Office.

Established analytical methods that are newly introduced at Frontier require performance evaluation prior to use with client samples. The QA Officer will require specific performance evaluation tests to be performed on a case-by-case basis. New methods that Frontier has developed require additional validation before client samples can be analyzed. Accuracy (bias), precision, and sensitivity will be assessed using QC samples with established control limits. When available, the developed method may be challenged in an intercomparison study with a promulgated method. The researcher may submit an article for publication in a peer-reviewed journal detailing the method and its limitations. Published articles provide recognition by the scientific community and is strongly encouraged by Frontier GeoSciences.

10.12 Control Charts

Frontier's LIMS database allows most QC results (LCS% Rec., LCS/LCSD RPD, MS% Rec., MS/MSD RPD, Dup RPD, ICV% Rec., CCV% Rec. and PBLKs) to be instantaneously collected in the form of control charts. Control charts allow the QA Office and laboratory staff to spot unfavorable analytical trends as they are developing. Corrective actions for those trends can in turn be assessed in real time. Additionally, control charts are periodically used in the calculation of efficiency factors for certain distillation and precipitation methods.

10.13 Data Reduction and Review

Data review and validation ensures that raw data is properly reduced and accurately transcribed to the correct reporting format. SOP FGS-038, "Data Validation," is referenced for data reduction and review. After the data has been acquired, data



reduction is performed using validated spreadsheets and LIMS databases that automate calculations as much as possible. Initially the data review is performed by the analyst, then the dataset is peer reviewed by another analyst, supervisor, or other staff trained in data review, who also have a strong understanding of the analysis. Peer-review consists of validating at least 5% of the calculations and 100% of the following:

- Transfer of raw data from the digestion sheet or bench sheet to the electronic spreadsheet and/or LIMS.
- Project name
- Data set ID
- sample identities
- Peak heights
- Instrument calibration
- All QC samples (e.g., blanks, CRM, ICV, CCVs, LCS, LCSD MD, MS and MSD)
- Detection and reporting limits
- Compliance with the individual method
- QC sample results must be reviewed for accuracy and precision as established by Frontier and/or client specifications
- Documentation of corrective actions and outliers

If the data meets all Frontier and contract mandated QC requirements, the raw data is stamped "Quality Assurance Peer-Reviewed" with the reviewer's initials and the date.

The LIMS status of the dataset is changed from analyzed to reviewed by the peer reviewer, making the sequence available for the Project Manager to generate the client report. The status of High QA projects are set to initial review after being peer reviewed. The data set is then logged into QA's "QA Dataset & Report Review Tracking Log." After High QA review the status in LIMS is changed from initial review to reviewed.

All reviewed datasets are scanned and saved as PDF files in: Quality Assurance_Recent Scanned Datasets

10.14 Report Format and Contents

In general, all reports consist of a the following sections: cover letter, analytical report of samples, case narrative, chain of custody forms, analytical results, matrix duplicates/triplicate results, matrix spike and matrix spike duplicate recovery and RPD, laboratory control sample/laboratory control sample duplicate recovery and RPD, preparation blanks, notes and definitions. High QA reports also include Initial and



Continuing Calibration Verification Standards (ICVs and CCVs) Initial and Continuing Calibration Blanks (ICBs and CCBs) and all analytical raw data. All non-compliant data (based on Frontier or contract-specific QC requirements) must be addressed in the case narrative section of all reports.

A standard Excel EDD file is generated and submitted to the client as well. Client specific EDD files can be generated by the IT department upon request. Frontier SOP FGS-080, "Electronic Reporting," details the procedures for electronic data deliverables (EDDs). All EDDs are peer reviewed by the Project Manager. To keep EDDs uniform and consistent, formatting and specifications of each client's EDD remain constant unless otherwise agreed (for project-specific EDDs). If errors are found before or after EDD submission, corrective action is taken.

If errors are found after the report has been submitted to the client material amendments to a test report are in form of a further document, or data transfer, which include the statement: "Supplement to Test Report for Work Order#" or an equivalent form of wording.



11 PERFORMANCE AND SYSTEM AUDITS

11.1 Internal Laboratory Audits

It is the responsibility of the Quality Assurance Officer to coordinate and conduct annual internal audits of the laboratory according to SOP FGS-041, "Internal Quality Assurance Audits" to verify that the activities continue to comply with the requirements of the quality system and NELAC 2003 Quality standards.

Shortly after the audit, the QA Officer writes an audit report with observations and findings and presents it to the Operation Manager and the Operations Management group. The Operations Manager, working closely with the Operation Management group, has two weeks to provide a written response to the report detailing corrective actions and implementation dates. If the QA Officer accepts the response, the audit report and response are validated and filed in the QA Office.

The laboratory shall notify clients within three business days, in writing, of any event such as the identification of defective measuring or test equipment that casts any doubt of the validity of the analytical results.

Follow-up audit activities shall verify and record the implementation and effectiveness of the corrective action taken.

11.2 External Laboratory Audits

Frontier views third-party audits as a form of consultation and welcomes the opportunity to improve the quality of our lab. On average, Frontier is audited approximately four times each year. External audits enable Frontier to qualify for and maintain accreditation through state governments and NELAP. Additionally, clients may audit us as part of a potential or ongoing contract. The QA Office maintains records of all such audits, their findings, and their corrective actions.

11.3 Management Committee Review of Quality Assurance

At the end of the year, the QA Officer prepares a QA Program Annual Report for Frontier's Operation Management group. This report presents a summary of QA issues from the previous year. It includes updates relating to certifications, audits, proficiency testing and inter-comparison results, control charts, and incident reports. The Operation Management group is required to review and validate all QA Program Annual Reports within two weeks of their release. If the Operation Management group provides no feedback to the QA Office during this time period, the report is considered validated by default. After the report has been validated, it is distributed to all members the Operation Management group. A file of past reports is maintained in the QA Office.



11.4 Corrective Actions for NELAC policies

There are times in the course of analytical laboratory work when there are departures from strict adherence to a particular lab policy defined by NELAC. The requirement is that all staff members work in a manner consistent with NELAC and the conformance with these policies is recognized. Our Quality Assurance Program tries to foster a creative environment. Bench level staff is instructed never to compromise data quality or safety over written instructions. However, departures must be documented appropriately, and all stakeholders must be informed. Reasons for a departure from written policy may include safety concerns, data quality, research and development, acts of nature (e.g., storms and power outages), matrix interference, and instrument performance.

SOP FGS-087, "Procedures for Deviation from Laboratory Policy", covers the procedures for intentional deviation from lab policy. Corrective actions may include filing an incident report, revising an SOP, revising the QAP, revising a safety procedure, or writing a new SOP. These deviations are considered an unusual situation and not a normal operating procedure. New methods need to be developed if problems are persistent.

11.5 Incident Reports

Mistakes and accidents occur in the course of analytical laboratory work. These must be immediately reported to the supervisor and documented on an Incident Report Form. If there are safety concerns, a report is also filed with the Health & Safety Officer.

An Incident Report Form is completed when a problem arises that requires a deviation from the applicable SOP or method. The deviation may be due to a mistake or accident. It also may be due to unforeseen problems with a sample, instrument or dataset. Whatever the circumstance, it must be recorded as soon as possible according to SOP FGS-039, "Incident Report Forms". It is the responsibility of each Group Supervisor (or delegate) to complete the Incident Report Forms and submit them to the QA group for review and follow-up. Completed Incident Report Forms are kept on file in the QA group and are assessed quarterly as part of each internal audit.

11.6 Customer Complaints

Project Managers and all other staff responding to customer complaints reference SOP FGS-097, "Customer Complaint Procedure and File." A customer complaint is defined as a complaint, concern, or question about data quality provided by Frontier or a subcontract laboratory. Complaints in customer service are not under the scope of this procedure. The QA Office keeps a file of customer complaints and Frontier's responses. This file is available to all clients for review except in cases where client confidentiality could be compromised.

The Project Manager generally handles all customer complaints. Clients may, at any time, request to discuss the matter with the Project Management Group Leader, the Operations Manager, or the President. The Project Manager may ask the Project Management Group Leader, the Laboratory Manager, or the President to help resolve



client complaints. The Project Manager is responsible for documenting the complaint and how it was resolved. This documentation is forwarded to the QA Office for filing.



12 DOCUMENT CONTROL AND MANAGEMENT

12.1 Document Management for Raw Data and Reports

After review and validation, original datasets are submitted to QA for filing. The dataset is recorded into the document management system by dataset ID and storage location of the original. Finally, all original datasets, chain of custody forms, generated reports, and log books are physically or electronically archived in a fireproof file cabinet, a fireproof vault, or an off-site storage unit, for a minimum of five years.

12.2 Document Management for Training Files

Training files for each technical staff member are maintained in the QA Office. It is the responsibility of each Group Supervisor to ensure that their staff has up-to-date training records and that these records are filed in the QA Office. Staff members are required to update their files with each revision of a training-related controlled document. Training files are examined as part of the internal auditing process.

12.3 Document Management for Controlled Documents

The QA Officer is responsible for ensuring that all controlled documents in use at Frontier reflect the most current revision. The QA Officer is also responsible for informing all Frontier staff when a controlled document has been retired in favor of a new revision.

SOPs and the QAP may be sent to individuals or entities outside Frontier in an "Uncontrolled Document" status. In such instances, Frontier is not required to inform the recipient of any changes or revisions to the document.

12.4 Document Management in the Event of Transfer of Ownership

In the event that Frontier GeoSciences transfers ownership, the legal transfer of documents will include the provision that the new owners will maintain all records according to the agreements established between the former Frontier owners and their clients.

If Frontier goes out of business, all records, to the best of Frontier's ability, will be transferred according to clients' instructions.



13 FACILITIES INVENTORY AND CONTROL

13.1 Facilities and Equipment

Frontier's 14,000 ft² research and analytical laboratory facilities are located in downtown Seattle, Washington. The location is close to Seattle-Tacoma International Airport, the University of Washington, and National Oceanic and Atmospheric Administration (NOAA). The space contains a large laboratory used for sample preparation, a 1,000 ft² bottle washing and storage area, and several Class-100 clean air stations.

Frontier has a dedicated sample shipping and receiving area (with Class-100 clean air station), Mercury Deposition Network (MDN) staging room, long-term sample storage trailer, hazardous waste control facilities, workshop, conference room, walk-in fireproof safe (where our server and computer backup system are kept), and staff offices. Frontier's entire space is a locked facility.

The laboratories are served by a custom-designed HVAC system, providing an atmosphere that is clean and well isolated from outside dust and dirt. Each laboratory atmosphere is monitored for gaseous mercury and appropriate action is taken if it exceeds 25ng/m³ in any location. Water systems are also checked weekly for trace metals content. Disposal of all other toxic materials is carried out under contract with a certified disposal company. The entire FGS space is periodically inspected for compliance with all city and state code requirements for fire, emissions, and storage of low-level radioactive samples.

The offices are equipped with document production equipment including laser printers, document and image-processing software, high volume photographic-quality color printer, large-capacity collating copiers, and a binding machine. A LAN connects staff computers and printers for local access, as well as providing external email, fax, and Internet access. Frontier also maintains a web site at www.FrontierGeosciences.com, and has a FedEx Powership shipping computer with access to FedEx pick-up as late as 5:00 PM Pacific time. The project Managers can make arrangements for staff to be on Saturdays to receive sample shipments.

Frontier Geosciences Inc., Capital Equipment for Analytical Use

Quantity Instrumentation:

- 12 Cold Vapor Atomic Fluorescence Hg Detector
- 4 Isothermal GC for Hg Speciation
- 2 Tekran 2537 Atmospheric Hg Monitor
- 2 Tekran 2600 Automated Aqueous Hg Analyzer
- 8 Ambient Air/Flue Gas Sampler



- 2 LUMEX Real Time Low Level Atmospheric Hg Detector
- 1 Perkin-Elmer ELAN-6000 ICP/MS
- 1 Perkin-Elmer Elan-6100 ICP/MS-DRC
- 1 Perkin-Elmer Elan-6100 ICP/MS-DRCII*
- 1 Perkin Elmer HPLC system 200 (*used for Se, As, and Cr speciation)
- 1 OI Analytical Alpkem FS300 Cyanide Analyzer
- 1 Cryotrap-HG/AAS System (As speciation)
- 1 IC-HG/AFS System (Se speciation)
- 1 UV-VIS Spectrophotometer
- 1 PSA "Millennium" HG/AFS System (As, Se, Sb)
- 1 Low-Level Ozone Analyzer/Calibrator
- 3 Specific Ion/Conductance/pH Meter
- 14 Class-100 Clean Air Hood
- 4 Milli-Q Reagent Water System
- 1 4' Hg-Free Nitrogen-Purge Glove Box
- 2 Large Volume Centrifuge (250-mL bottle)
- 6 Methyl Hg Distillation Units
- 5 Complete sets of Fluegas Sampling Equipment
- 24 Teflon Bulk Deposition Collectors
- 2 Gold Sputter Coater
- 6 Digital Mass Flow meter
- 2 Teflon-Coated Go-Flo Water Sampling Bottle
- 2 Ultra-Clean Peristaltic Field Sampling Pumps
- 1 Temperature Controlled Gas Reaction Mixing System
- 1000's Ultra-Clean Teflon Bottles (various sizes)



13.2 Security

Access to Frontier offices and laboratories is regulated and limited to authorized personnel. All outside doors are kept locked at all times. Visitors must first press the access button on the building's security keypad outside the main entrance, identify themselves, and then be admitted by an employee-actuated electronic door lock release. Visitors are required to check in and sign the guest list on arrival, and to sign out on departure. Regular visitors may be allowed unescorted access to general areas of the building, provided that their names and other details are recorded at the front desk. All visitors in laboratory areas must be accompanied by a Frontier employee.

Employee safety is an important concern at Frontier. In addition to the overall facility security system, Frontier also has two remote alarms. The alarms are worn around the neck, and alert the police when activated. Laboratory security is checked routinely as part of Frontier's internal audit program.

13.3 Computer Systems and Software

FGS employs seven servers, all running Windows Server 2003 in an Active Directory environment. Two of the seven servers act as domain controllers to control and maintain security access and access policies using the NTFS file system. Separate servers are utilized to control remote access to the network, terminal services, file and print services as well as the company Exchange 2003 email server. In addition to the Windows 2003 servers FGS employs one additional server running VMWare ESX Server 3.5.1. All servers are equipped with backup drives and appropriate backup software that provides scheduling, automation, and monitoring of back-ups.

The server room is located in a cement-lined vault with a 6-inch thick metal door. It is closed each evening according to the lab lockup procedure to protect against fire. There is a facility-wide security system with motion detection that is activated each evening, also according to the lockup procedure to protect against theft. The servers and other network hardware are installed at least three inches above the floor to protect from water damage.

Each server is attached to a UPS system with monitoring software and has enough battery power to keep the server running for at least twenty minutes. If the power is out for more than five minutes, the software will shut down the server automatically storing all data before battery power runs out. Network devices such as routers, switches, and hubs are also attached to a UPS device. All other FGS computers also have some form of UPS to minimize data loss, and loss of instrument control due to a short power failure. The servers, network hardware, and all other FGS computers' AC power supplies are plugged into power strips with built-in surge/spike protection.

Symantec Endpoint Protection antivirus software with immediate file protection services is installed on each server. Files on the server disks are scanned daily. Virus definitions are updated automatically each day via an Internet connection to the software vendor. Symantec Endpoint Protection for Exchange is installed on the mail server to scan incoming and outgoing mail attachments for viruses. Attachments with the file extensions ".exe", ".pif", ".bat", ".vbs", and ".scr" are deleted from all e-mails that are



sent to FGS. E-mail alerts are sent to IT personnel upon detection of viruses or unauthorized attachments. If FGS gets a large number of infected attachments in a day, the Internet mail service may be shut down until the problems are rectified. Symantec Endpoint Protection is also installed on each employee's computer to protect against infected files brought in through the Internet, outside e-mail accounts, or portable diskettes, and flash drives.

Access to computers and files is limited to domain users with passwords that grant access to job-specific files and folders using the file securities built into the file system. Data security has been divided into three categories: access, protection against corruption, and redundancy. Access to data is subject to levels of control. The data owner determines data criticality. Non-critical data is available throughout the network. Critical data is available to members of predefined groups only. Sensitive and proprietary data is restricted at the user level. Data is protected from corruption by a strategy of limited access and redundancy. Redundancy takes the form of data backups via computer and secure storage of data in hard copy. Backups cover the primary domain controller, the backup domain controller, and individual workstations.

13.4 Bottle and Acid Vat Monitoring

Ensuring that our sample collection containers are appropriately cleaned for ambient water sampling is vitally important to Frontier. A bottle monitoring program gives us quantitative evidence that our procedures are contaminant-free. Records of each test performed under the bottle monitoring program are maintained by the QA Office and are available upon request.

Purchased glass bottles and vials for mercury analysis are sequestered by lot from a vendor. Prior to use and distribution of glass bottles and vials for mercury analysis, 5% of the lot are randomly chosen and tested for total mercury to ensure that the containers are free of contamination. When contamination is identified, alternative lots are tested until a lot free of contamination is found. The random testing procedure is repeated when a change in lot number occurs.

Cleaned Teflon and polyethylene bottles are monitored monthly by random testing of cleaned bottles. Each month, 5% of the total amounts of bottles cleaned are tested to identify potential bottle cleaning sources of contamination. When contamination is identified, the cleaning process is repeated until no further contamination is identified. Only bottles that have been found to be free of contamination are shipped to our clients.

13.5 Air Monitoring

Frontier's mercury analyses require ultra low levels of mercury in laboratory air. All laboratories at Frontier are monitored for mercury contamination by direct measurements with LUMEX real time low level atmospheric Hg detector. The action limit for laboratory air is 25ng/m³. If a laboratory exceeds the action limit, air flow is increased and the location is monitored until levels agrees with previous collected back ground data from this location. Records of each test are maintained by the QA Office and are available upon request.



13.6 Reagent Water Monitoring

Ensuring that our reagent water is free of contamination sources for sampling and analysis of ambient water is critical to the success of Frontier's laboratory facilities. Our reagent water monitoring program gives us additional quantitative evidence that our procedures are contaminant-free. Records of each test performed under the reagent water monitoring program are maintained by the QA Office and are available upon request.

All reagent water is monitored for a variety of analytes on a weekly basis. Acceptable results from these tests confirm each water system's suitability for analytical use. If a system produces unacceptable results, it is sequestered until subsequent analyses verify freedom from contamination. Control limits and records of each test are maintained by the QA Office and are available upon request.



14 REFERENCES

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Appendix 1:

Frontier GeoSciences Organizational Chart

As of July 2008

